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THE DETERMINATION OF THE COMPOSITION OF SLURRIES BY THE MEASUREMENT OF THERMAL-NEUTRON-CAPTURE GAMMA RADIATION

H. P. DIBBS, J. L. DALTON AND S. S. NARGOLWALLA

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THE DETERMINATION OF THE COMPOSITION OF SLURRIES BY THE MEASUREMENT OF THERMAL-NEUTRON-CAPTURE GAMMA RADIATION

by

H. P. Dibbs*, J. L. Dalton** and S. S. Nargolwalla***

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ABSTRACT

A study has been made of the application of thermal-neutron-capture gamma radiation to the determination of the composition of aqueous slurries of iron oxide, cupric oxide, zinc oxide, sulphur, and silica. In general, a linear relationship was found between the solids content of the slurry and the prompt-gamma-ray counts from hydrogen and from the solid phase, and an equation is presented that predicts the variation of the prompt-gamma-ray counts from hydrogen with the composition of the slurry.

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LA DÉTERMINATION DE LA COMPOSITION DES COULIS EN MESURANT LE RAYONNEMENT GAMMA POUR LA CAPTURE DE NEUTRONS THERMIQUES

par
H. P. Dibbs*, J. L. Dalton** et S. S. Nargolwalla***

RÉSUMÉ

Les auteurs ont fait une étude de l'application du rayonnement gamma pour la capture de neutrons thermiques pour déterminer la composition des coulis aqueux d'oxyde de fer, d'oxyde cuivrique, d'oxyde de zinc, de soufre et de silice. En général, ils ont trouvé qu'il y avait une relation linéaire entre la teneur en solides du coulis et les comptes de rayons gamma instantanés de l'hydrogène et de la phase solide, et les auteurs présentent une équation qui prédit la variation des comptes de rayons gamma instantanés de l'hydrogène avec la composition du coulis.

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CONTENTS

TABLES

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INTRODUCTION

The measurement of thermal-neutron-capture gamma radiation has attracted considerable interest in recent years for the non-destructive determination of major elements in bulk samples[1-6]. The basis of the method is that capture of a thermal neutron (0.025eV) by a nucleus gives a highly unstable compound nucleus which loses energy rapidly (10^{-14} to 10^{-15} seconds) by the emission of prompt-gamma radiation. This (n, γ) process is shown in Equation 1,

$$ {}_{z}X^{A} + n \rightarrow \left[{}_{z}Y^{A+1} \right] \rightarrow {}_{z}Y^{A+1} + \gamma \qquad \ldots \text{(Eq. 1)} $$

where ${}_{z}X^{A}$ is the target nucleus, $\left[{}_{z}Y^{A+1} \right]$ the compound nucleus, and ${}_{z}Y^{A+1}$ the product of the neutron reaction which may be stable, or radioactive. In the latter case, the product nucleus will have a characteristic half-life, and will decay with the emission of beta and/or gamma radiation which may be detected by conventional counting techniques. The energy of the gamma radiation emitted on neutron capture is essentially that of the binding energy of the neutron in the compound nucleus and, except for a few low atomic-number elements, is about 5 to 8 MeV. The decay of the compound nucleus to the ground state usually occurs through several intermediate energy states to give a gamma-energy distribution, characteristic of the target nucleus, that may be used for its identification and quantitative measurement. Because of the high energy of prompt-gamma radiation (Table 1), virtually no interference is experienced in its spectral analysis from the lower energy (usually <2 MeV) gamma radiation emitted in the decay of radioactive nuclei or in neutron inelastic-scattering reactions[7-9].

TABLE 1

Nuclear Properties of Some Elements for Prompt-Gamma-Ray Analysis

Element	Thermal Neutron Cross Section (barns)	Principal Prompt-Gamma-Peaks (MeV)
H	0.335	2.23
O	<0.0002	--
Si	0.16	3.54, 4.93, 6.38
S	0.52	3.22, 5.42
Fe	2.62	7.65, 7.63
Ni	4.6	6.84, 8.53, 8.99
Cu	3.85	7.63, 7.91
Zn	1.10	7.86
W	19.1	6.19, 5.32, 5.26
Pb	0.17	7.37

The present interest in the gamma-ray-capture technique arose from measurements[10] of the water content of iron oxide slurries by the thermalization of high-energy neutrons from a 1.1 mCi Ra/Be source (output, 1.2×10^4 neutrons/sec). In this study, a linear relationship was found between the output of the thermal-neutron detector and the weight of iron oxide in the slurry. It was felt that a similar experimental arrangement, in which a NaI(Tℓ) detector was substituted for the thermal-neutron detector, should permit the simultaneous determination of hydrogen and other elements in a slurry and provide an analytical method that would have applications to process control in the minerals industry. It should be noted that an aqueous slurry has a large hydrogen content and, therefore, is an excellent thermalizing medium[10] for the high-energy neutrons emitted from isotopic neutron sources (Table 2) used in prompt-gamma-ray analysis.

The main objective of the present study was to obtain comparative sensitivity data for selected elements of economic importance, so, for this reason,elements or oxides were used in the tests; oxygen produces no interference in prompt-gamma-ray measurements because of its very low neutron-capture cross section (Table 1).

TABLE 2

Characteristics of Some Isotopic Neutron Sources[10]

Source	Type	Half-Life	Neutron Output (n/sec/Ci)	Average Neutron Energy (MeV)
Ac-227/Be	(α,n)	28.1 yr	2.0×10^7	5
Am-241/Be	(α,n)	458 yr	2.2×10^6	4
Cm-242/Be	(α,n)	163 d	3.0×10^6	4
Ra-226/Be	(α,n)	1620 yr	1.3×10^7	3.6
Th-228/Be	(α,n)	1.91 yr	2.0×10^7	4
Cf-252	Spontaneous Fission	2.65 yr	4.4×10^9	2.3

EXPERIMENTAL METHOD AND RESULTS

The prompt-gamma probe (Figure 1) was designed for bore-hole logging studies and contained a 1-Curie Am/Be neutron source (output, 2.5×10^6 neutron/sec) and a 2 x 2-inch NaI(Tℓ) scintillation detector which were mounted in a Lucite tube (O D 5.75 inches,. 48 inches long). The neutron source was surrounded by paraffin wax to thermalize the fast neutrons, and a lead "shadow-shield" was placed between the source and the detector to shield it from direct radiation from the source. Borated-wax was also placed around the detector to limit the thermal neutron field in its immediate vicinity. The probe was placed in a closely fitting Lucite tube and mounted in a 60-gallon polyethylene tank (Figure 1).

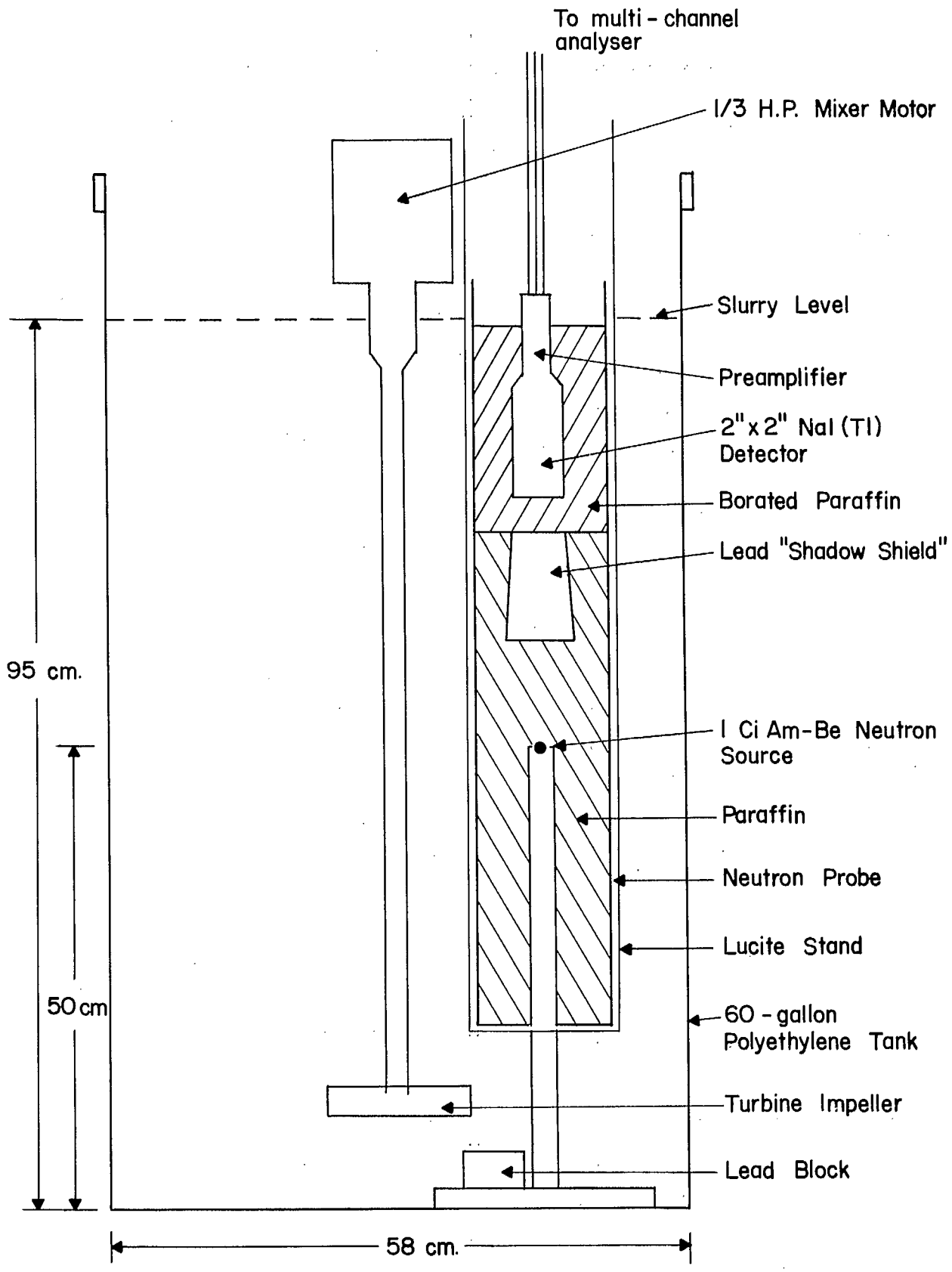

FIGURE 1. SCHEMATIC DIAGRAM OF EQUIPMENT FOR MEASURING PROMPT-GAMMA RADIATION FROM SLURRIES

Initial tests were made to determine the effective measurement volume of the prompt-gamma probe by adding successive amounts of water to the tank and measuring the prompt-gamma-ray counts from hydrogen after each addition. Counts were taken with the probe positioned at the centre of the tank and at a point mid-way between the centre and the wall of the tank, and the results are shown in Figure 2. The first position represents optimum geometry for this particular system, i.e., with equal amounts of water on all sides of the neutron source, and the second position, of somewhat lower detection efficiency, is the one which had to be used for the slurry measurements to allow space above the centre of the tank for the stirrer motor[10]. In both positions, a maximum count-rate was obtained with the water at about the 95-cm level.

Measurements were made of the prompt-gamma emission from slurries of iron oxide, cupric oxide, zinc oxide, sulphur, and silica. The iron oxide was a minus 320-mesh concentrate from the Quebec Cartier Mining Company, Gagnon, Quebec, that contained 69.6 per cent iron; the other materials were technical-grade reagents of greater than 98 per cent purity. A small amount of a surfactant (Tween 20, Fisher Scientific Limited) was added to the water tank to assist slurry formation in the sulphur run; the other materials slurried readily.

The gamma-ray spectra and data acquisition were obtained with a 4096-channel pulse-height analyser (Canberra Industries Limited) whose gain was stabilized on the hydrogen prompt-gamma peak (2.23 MeV). In performing a slurry run, the tank was first filled to the 95-cm level with water and the prompt-gamma-ray spectrum from 1.5 to 10 MeV was recorded. Serial additions of 15 to 20 pounds of the solid were then made, and, following each addition, the gamma-ray spectrum was measured over the same energy range. Because the prompt-gamma probe had a low detection efficiency, impracticably long counting times would have been required to obtain spectra with well-defined peaks. Therefore, integral counting was performed in the slurry tests and net counts were obtained,

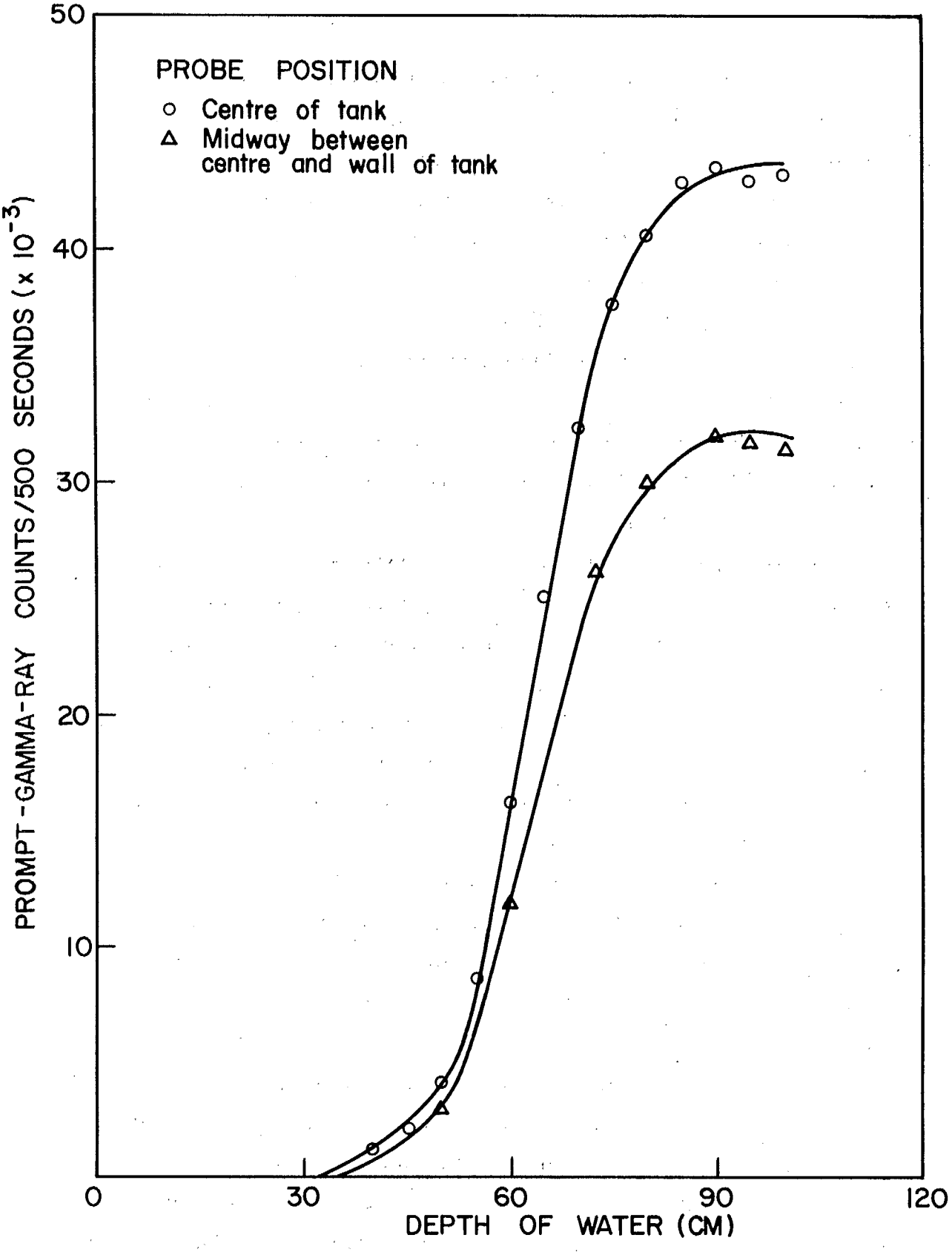

FIGURE 2. VARIATION OF PROMPT-GAMMA-RAY COUNTS FROM HYDROGEN WITH DEPTH OF WATER IN SLURRY TANK

over selected energy intervals appropriate[11] to the element in the slurry, by subtracting the background counts in the same energy intervals with only water in the tank. The hydrogen prompt-gamma peak was well-defined in all the runs, and the net counts under its peak were obtained.

The net prompt-gamma-ray counts for some of the spectral regions investigated are shown as a function of slurry density* for iron oxide, cupric oxide, zinc oxide, and sulphur in Figures 3 to 6. The counts under the 2.23 MeV hydrogen peak are given for iron oxide slurries, in Figure 7, and for slurries of cupric oxide, zinc oxide, and sulphur in Figure 8. The most extensive investigation was made with slurries of iron oxide, and a linear regression analysis of the data in Figures 3 and 7 is given in Table 3. With slurries containing up to 200 lb silica, only small changes were found in the prompt-gamma-ray spectra from that of a blank run with water. However, with a high sensitivity prompt-gamma probe, silicon may be determined unambiguously in a silica slurry[12].

Some prompt-gamma measurements were also made with coarse powders of nickel oxide (sinter grade) and lead oxide (technical grade). It was not possible to form a slurry with these samples, but a guide to the energy distribution of their prompt-gamma-ray spectra was obtained by loading each oxide into four 2 x 45-inch polyethylene tubes which were then positioned in a symmetrical array around the probe, the tank being filled with water. The results are given in Table 4.

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*Slurry density in wt % solids = $\frac{\text{Weight of solids} \times 100}{\text{Weight of water + weight of solids}}$

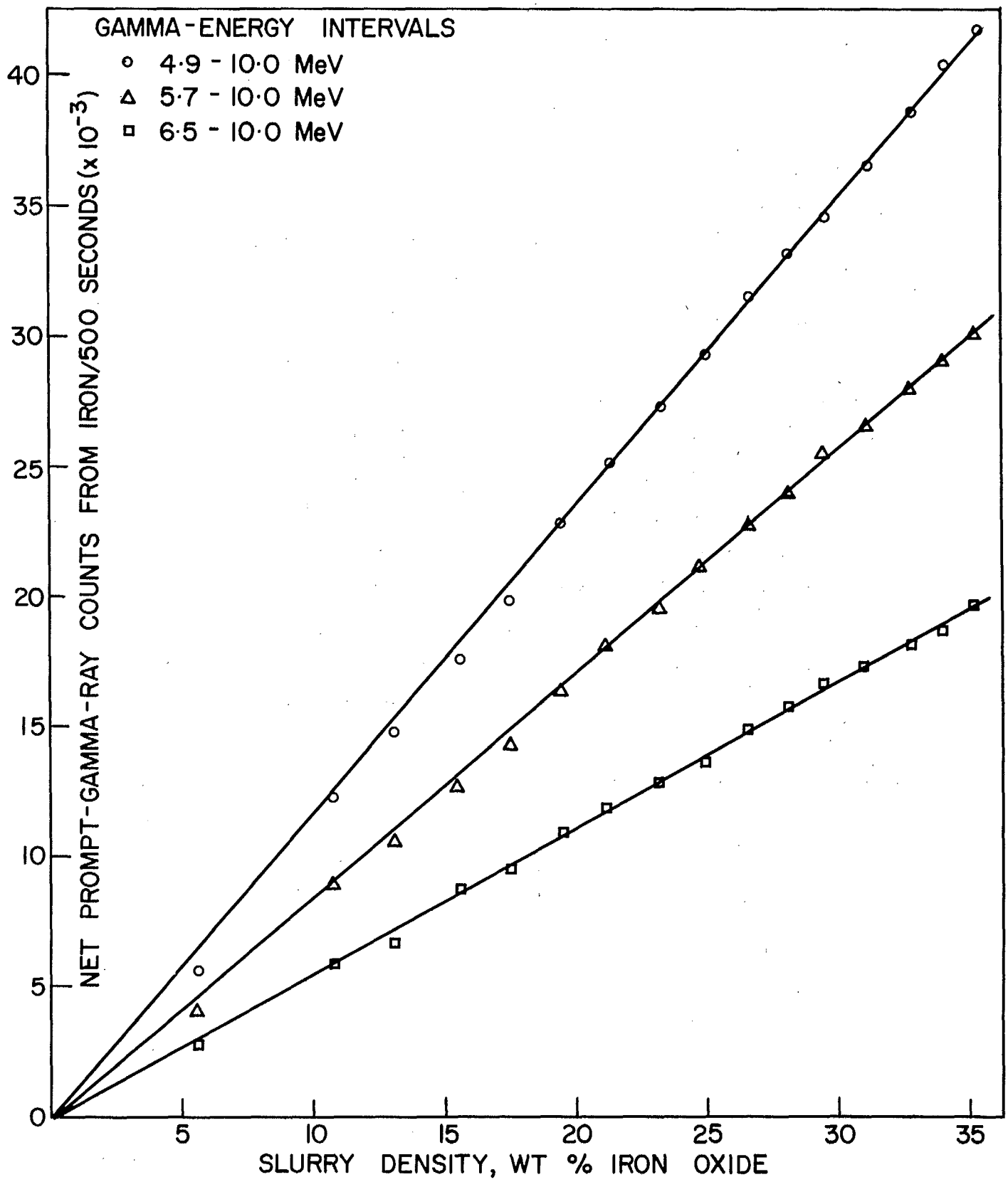

FIGURE 3. VARIATION OF PROMPT-GAMMA-RAY COUNTS FROM IRON WITH SLURRY DENSITY, FOR DIFFERENT ENERGY INTERVALS

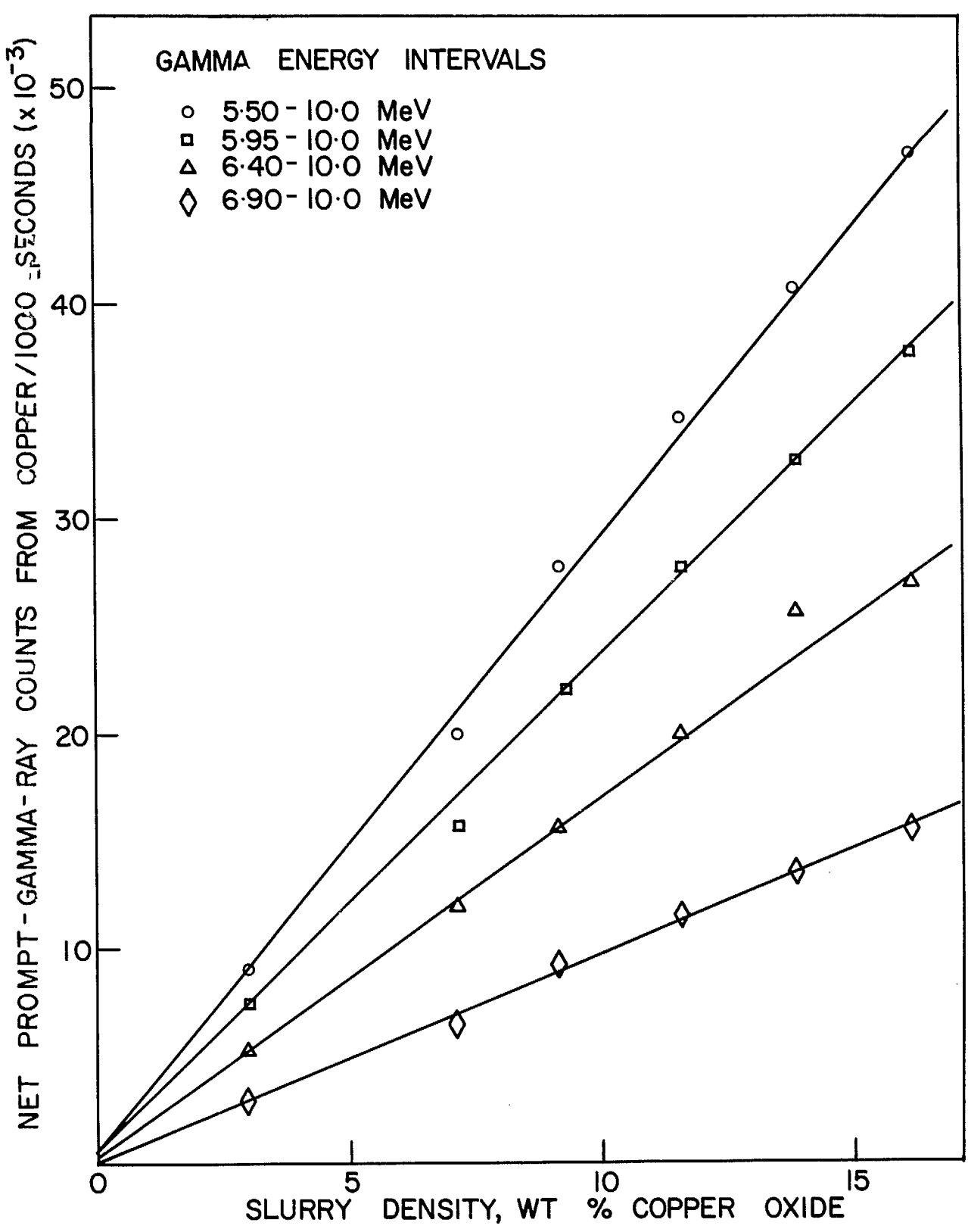

FIGURE 4. VARIATION OF PROMPT-GAMMA-RAY COUNTS FROM COPPER WITH SLURRY DENSITY FOR DIFFERENT ENERGY INTERVALS

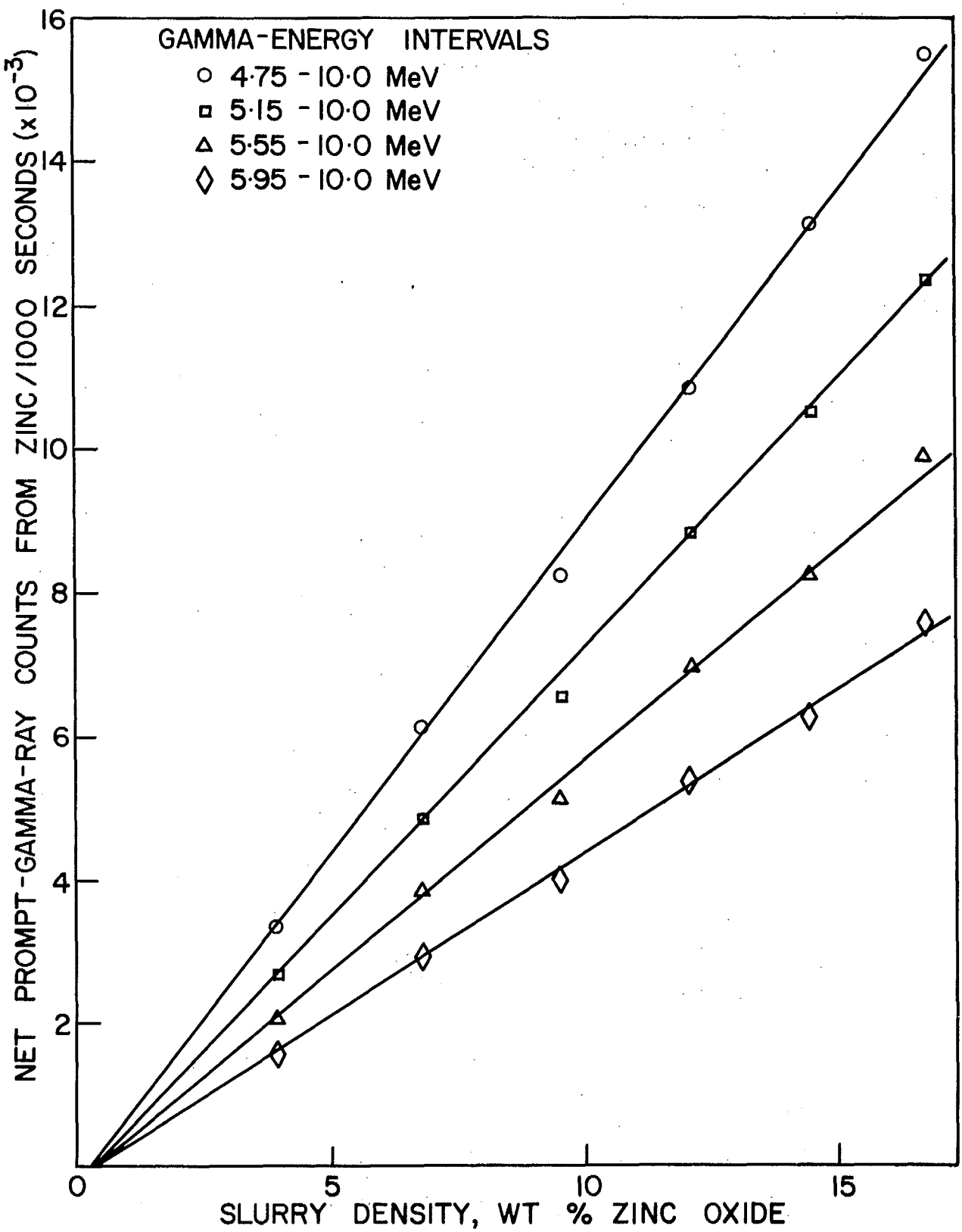

FIGURE 5. VARIATION OF PROMPT-GAMMA-RAY COUNTS FROM ZINC WITH SLURRY DENSITY FOR DIFFERENT ENERGY INTERVALS

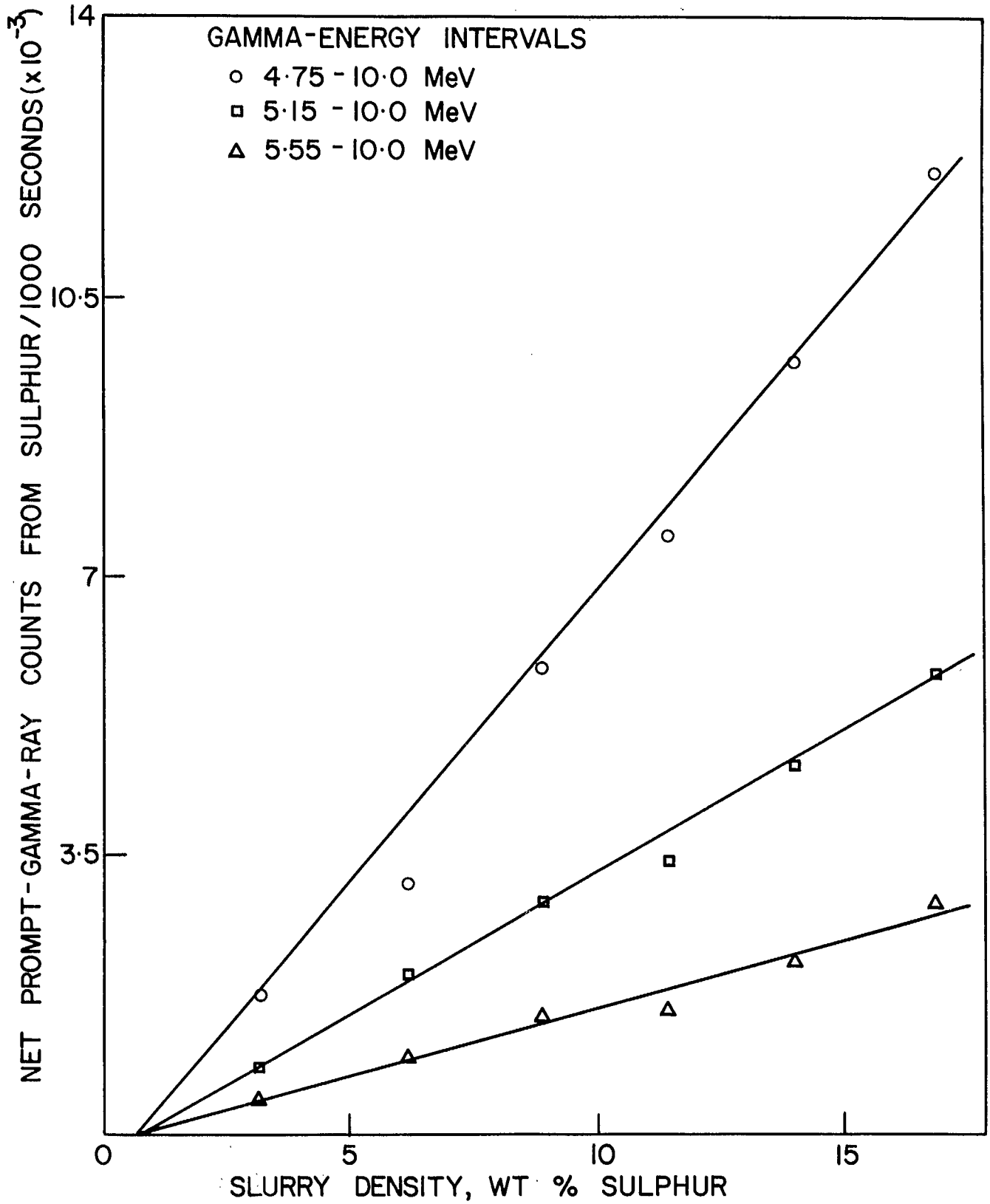

FIGURE 6. VARIATION OF PROMPT-GAMMA-RAY COUNTS FROM SULPHUR WITH SLURRY DENSITY FOR DIFFERENT ENERGY INTERVALS

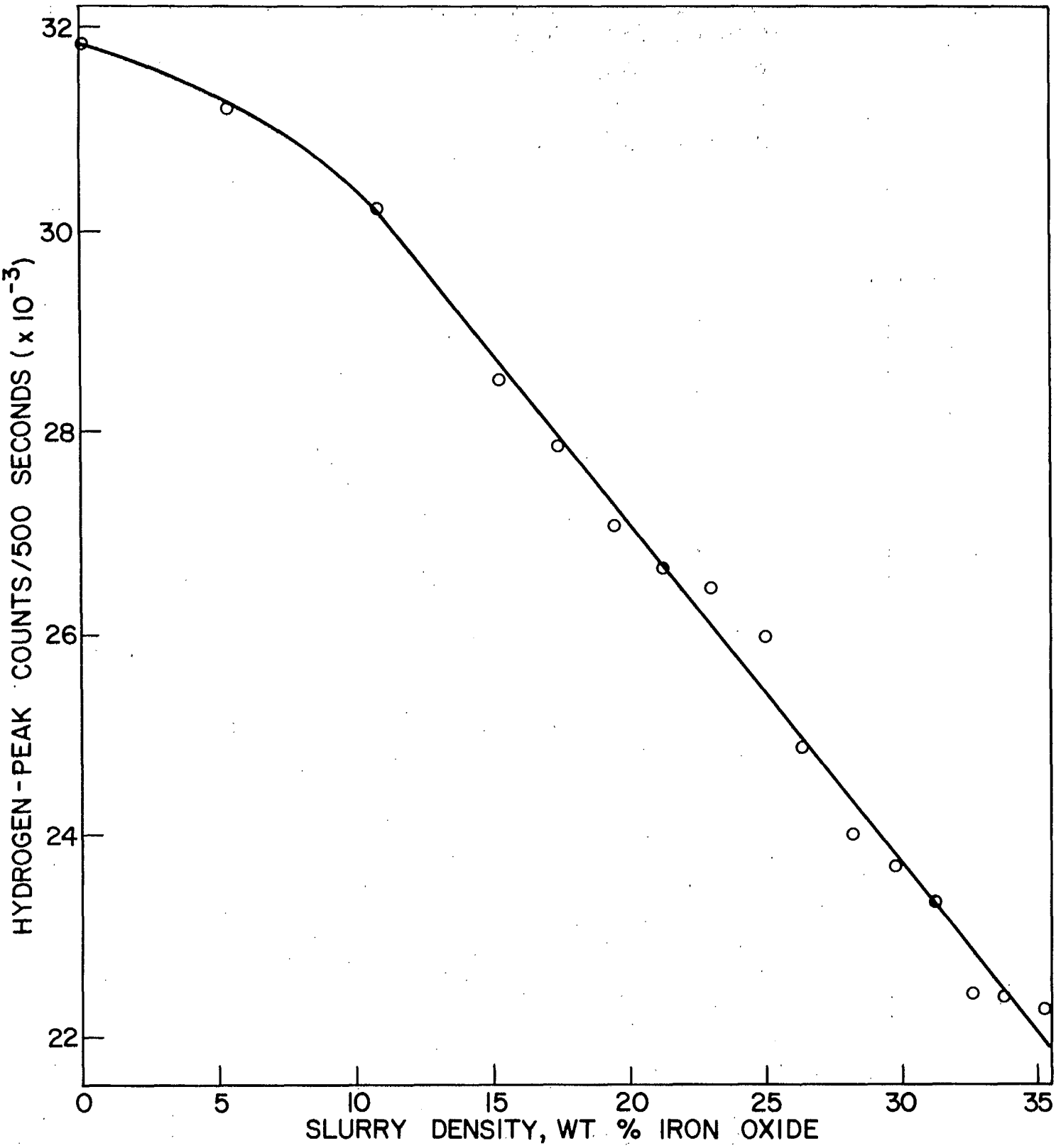

FIGURE 7. VARIATION OF PROMPT-GAMMA-RAY COUNTS FROM HYDROGEN WITH SLURRY DENSITY

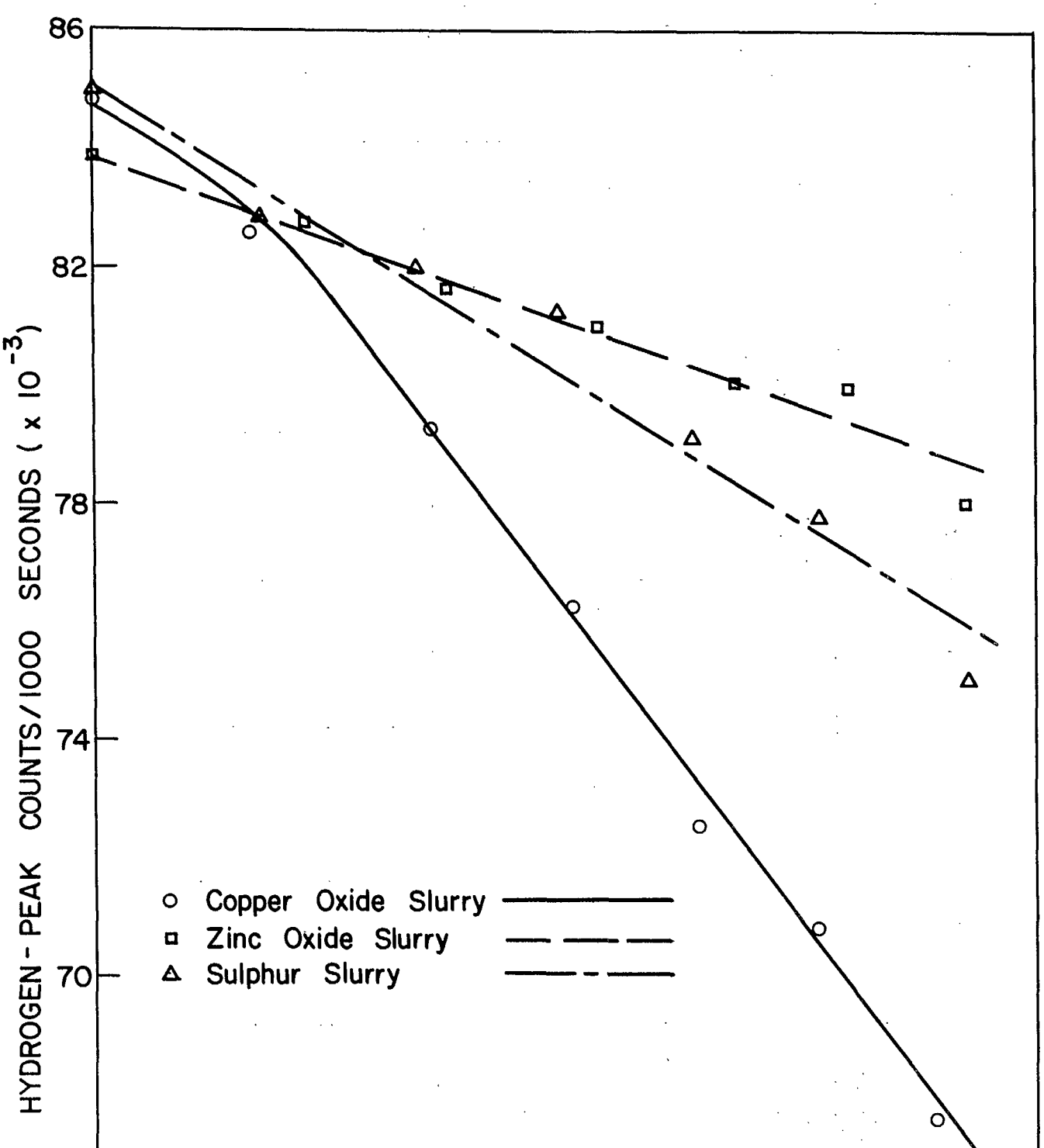

FIGURE 8. VARIATION OF PROMPT-GAMMA-RAY COUNTS FROM HYDROGEN WITH SLURRY DENSITY FOR COPPER OXIDE, ZINC OXIDE, AND SULPHUR

TABLE 3

Linear Regression Analysis of Prompt-Gamma-Ray Data for Iron Oxide Slurries

Item	Hydrogen Peak (MeV)	Energy Regions in Iron Spectrum (MeV)						
	2.23	4.8-10	5.7-10	6.5-10	6.7-10	6.9-10	7.1-10	7.3-10
Correlation Coefficient	-0.99031	0.99978	0.99980	0.99934	0.99890	0.99502	0.99775	0.99496
Intercept (Counts)	33,926	-1211.0	-781.92	-407.70	-0.88121	1142.6	182.35	707.47
Slope (Counts/%)	-342.40	1220.0	874.64	568.78	451.49	317.06	255.00	161.35
Standard Error (Counts)	370.85	234.93	161.90	189.52	193.53	290.23	156.60	148.72
Relative Standard Error at $\bar{x}$ (%)	1.4593	0.87176	0.83426	1.4000	1.8572	3.4302	2.5806	3.3558
Range of Solids Content (wt %)	10-35	5-35	5-35	5-35	5-35	5-35	5-35	5-35

TABLE 4

Prompt-Gamma-Ray Data for Nickel Oxide and Lead Oxide

Material	No. of Tubes	Hydrogen Peak Counts	Energy-Interval Counts (MeV)				
			5.5-10	5.95-10	6.42-10	6.90-10	7.40-10
Nickel Oxide	1	89,046	7,870	6,630	5,626	4,470	2,362
	2	87,406	13,236	11,212	9,514	7,584	5,350
	4	85,042	27,018	22,830	18,980	15,132	11,390
Lead Oxide	4	89,650	905	732	443	185	66

Counting Time: 1000 seconds.

Weight: 16 lb per tube.

DISCUSSION

It may be noted from Figures 7 and 8 that the slopes of the lines relating the prompt-gamma-ray counts from hydrogen to the slurry density depend on the solid component of the slurry. By assuming that thermal-neutron capture reactions provide the only mechanism by which neutrons are absorbed in the slurry, it is possible to derive (see Appendix 1) a simple relationship between the slurry density and the ratio (f_σ) of the prompt-gamma counts from hydrogen in a given volume of slurry (P_s) to the prompt-gamma counts from hydrogen in the same volume of water (P_w),

$$\frac{P_s}{P_w} = f_\sigma = \frac{\left(\frac{W}{M_w} \times 2\sigma_H\right)}{\left(\frac{W}{M_w} \times 2\sigma_H\right) + \left(\frac{S}{M_s} \times Y \times \sigma_A\right) + \left(\frac{S}{M_s} \times Z \times \sigma_B\right)} \quad \ldots \text{(Eq.2)}$$

where W = weight of water in the slurry,

S = weight of solid in the slurry,

M_w = molecular weight of water,

M_s = molecular weight of the solid,

A_yB_z = generalized formula of the solid phase,

σ_H, σ_A and σ_B = thermal-neutron absorption cross-sections of hydrogen, and elements A and B.

Plots of f_σ against slurry density are given in Figures 9 and 10 for slurries of iron oxide, cupric oxide, zinc oxide, and sulphur; the dashed lines give the relationship predicted by Equation 2 and the points are experimental values. Satisfactory agreement is found between the predicted and the experimental values of f_σ for iron, zinc, and copper oxides. The agreement for sulphur is poor and could be due to air, which was not displaced by water, adhering to the naturally hydrophobic surface of sulphur and to cause an apparent decrease in the hydrogen density in the slurry.

A linear relationship was also found between the prompt-gamma-ray counts from the solid phase in the slurry and the slurry density (Figures 3 to 6), and it is apparent from these results that simple prompt-gamma-ray analysis is feasible for a slurry that contains only two elements of significance from the nuclear viewpoint, e.g., the simultaneous determination of hydrogen (H_2O) and iron (Fe_2O_3) in an iron oxide slurry. By using a more intense neutron source and a larger NaI(Tℓ) detector, the sensitivity of these measurements could be increased, by two to three orders of magnitude, to provide excellent counting statistics in a short time (~10 seconds). The prompt-gamma-ray spectrum of a multi-element slurry will consist of the sum of the spectra of the individual elements and may be resolved, for a NaI(Tℓ) detector, by a computer-based method[13], or the contribution from each element may be measured directly with a high-resolution Ge(Li) detector[14]. From the current work, an appreciation of the relative prompt-gamma counts in different energy intervals for slurries of iron, copper, and zinc oxides and for sulphur may be obtained from Tables 5 to 8. These data have been normalized to a counting time of 1000 seconds and to 40 kg of the element.

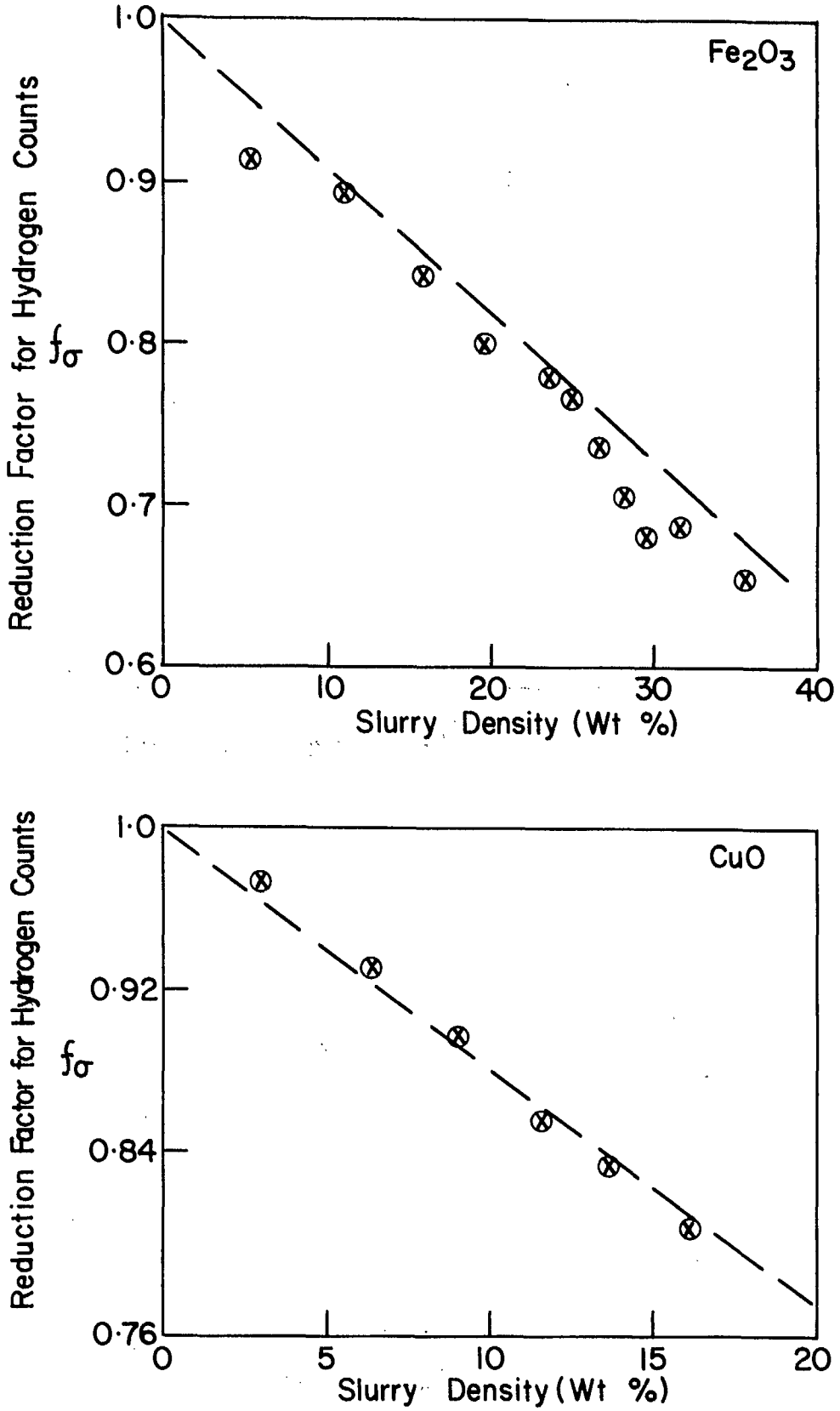

Figure 9. A Comparison of the Theoretical and Experimental Values of the Reduction Factor for Hydrogen Prompt-Gamma-Ray Counts with Slurry Density for Slurries of Iron Oxide and Cupric Oxide (--- theoretical; ⊗, experimental points).

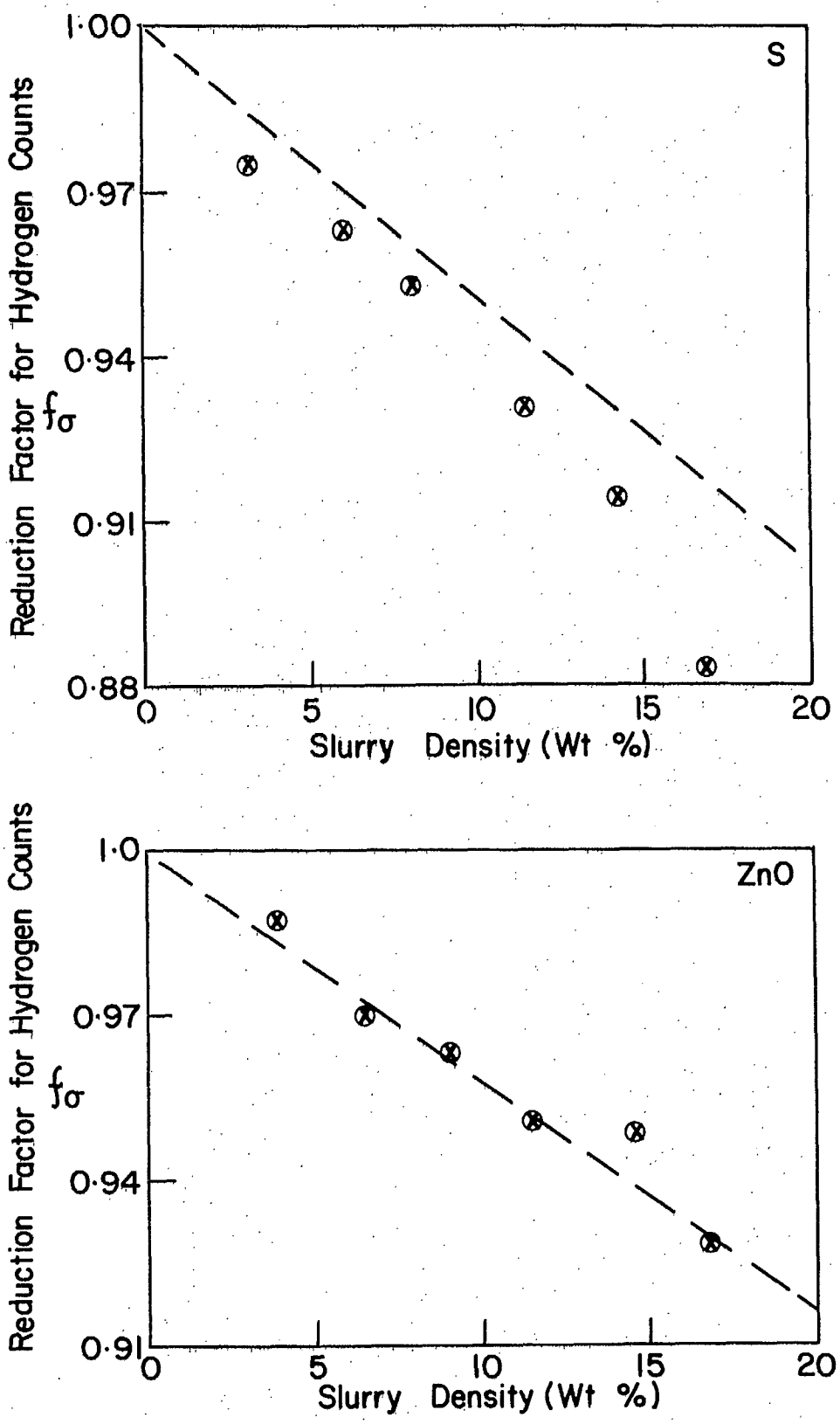

Figure 10. A Comparison of the Theoretical and Experimental Values of the Reduction Factor for Hydrogen Prompt-Gamma-Ray Counts with Slurry Density for Slurries of Sulphur and Zinc Oxide (--- theoretical; ⊗, experimental points).

TABLE 5

Prompt-Gamma Counts from Iron, in Different Energy Intervals, in a Slurry of Iron Oxide

Energy Interval (MeV)	4.80-10	5.70-10	6.50-10	6.70-10	6.90-10	7.10-10	7.30-10
Counts/1000 sec/40 kg Fe	50,160	36,132	23,700	19,556	15,950	11,355	6,968
Energy Interval (MeV)	4.80-5.70	5.70-6.50	6.50-6.70	6.70-6.90	6.90-7.10	7.10-730	
Counts/1000 sec/40 kg Fe	14,028	12,432	4,144	3,606	4,595	4,387	

TABLE 6

Prompt-Gamma Counts from Copper, in Different Energy Intervals, in a Slurry of Copper Oxide

Energy Interval (MeV)	5.50-10	5.95-10	6.40-10	6.85-10	7.30-10	7.75-10	8.30-10
Counts/1000 sec/40 kg Cu	54,000	43,660	31,660	18,000	7,045	1,410	70
Energy Interval (MeV)	5.50-5.95	5.95-6.40	6.40-685	6.85-7.30	7.30-7.75	7.75-8.30	
Counts/1000 sec/40 kg Cu	11,660	12,000	13,660	10,955	5,635	1,340	

TABLE 7

Prompt-Gamma Counts from Zinc, in Different Energy Intervals, in a Slurry of Zinc Oxide

Energy Interval (MeV)	4.75-10	5.15-10	5.55-10	5.95-10	6.35-10	6.75-10	7.15-10	7.55-10	7.95-10
Counts/1000 sec/ 40 kg Zn	17,100	13,850	11,820	8,400	5,895	3,935	2,141	953	394
Energy Interval (MeV)	4.75-5.15	5.15-5.55	5.55-5.95	5.95-6.35	6.35-6.75	6.75-7.15	7.15-7.55	7.55-7.90	
Counts/1000 sec/ 40 kg Zn	3,250	2,030	3,420	2,505	1,960	1,704	1,188	569	

TABLE 8

Prompt-Gamma Counts from Sulphur, in Different Energy Intervals, in an Elemental Sulphur Slurry

Energy Interval (MeV)	4.75-10	5.15-10	5.55-10	5.95-10	6.35-10	6.75-10	7.15-10	7.55-10	7.90-10
Counts/1000 sec/ 40 kg S	10,500	5,075	2,240	1,923	1,588	1,259	838	450	365
Energy Interval (MeV)	4.75-5.15	5.15-5.55	5.55-5.95	5.95-6.35	6.35-6.75	6.75-7.15	7.15-7.55	7.55-7.90	
Counts/1000 sec/ 40 kg S	5,425	2,835	317	835	329	421	388	85	

SUMMARY

This preliminary investigation indicates that thermal-neutron-capture gamma-ray analysis offers significant promise as a technique for the determination of a number of elements in slurries and should be well-suited to the mineral processing industry where large-volume samples are available. Because of the instantaneous emission of gamma radiation following neutron capture, the method is independent of flow-rate. The design of the probe used in these studies was by no means optimum for slurry measurements. Considerably better sensitivity could be achieved with a more intense neutron source and a larger NaI(Tℓ) detector, and such a system would be of value in investigating the effects of inter-element interference in the gamma-ray spectra from practical slurries.

ACKNOWLEDGEMENTS

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APPENDIX 1

The Effect of the Solid Phase in a Slurry on the Prompt-Gamma-Ray Counts from Hydrogen

Consider a slurry containing "W" grams of water, and "S" grams of a solid of density ρ. The volume of the slurry (V_S) in cm^3 is

$$V_S = \left(\frac{S}{\rho} + W\right), \qquad \ldots \text{(Eq. 1)}$$

and the number of hydrogen atoms per cm^3 (N_H) in the slurry is given by

$$N_H = \frac{\left(\frac{W}{M_W} \times N_O \times 2\right)}{V_S}, \qquad \ldots \text{(Eq. 2)}$$

where N_O is Avogadro's number and M_W is the molecular weight of water.

Similarly, for a solid of molecular weight M_S and general formula $A_Y B_Z$, the number of atoms A and B per cm^3 of slurry is given by Equations 3 and 4.

$$N_A = \frac{\left(\frac{S}{M_S} \times N_O \times Y\right)}{V_S} \qquad \ldots \text{(Eq. 3)}$$

$$N_B = \frac{\left(\frac{S}{M_S} \times N_O \times Z\right)}{V_S} \qquad \ldots \text{(Eq. 4)}$$

The macroscopic absorption cross-sections for hydrogen (Σ_H) and for elements A(Σ_A) and B(Σ_B) in the slurry are given by Equations 5 to 7,

$$\Sigma_H = N_H \sigma_H \qquad \ldots \text{(Eq. 5)}$$

$$\Sigma_A = N_A \sigma_A \qquad \ldots \text{(Eq. 6)}$$

$$\Sigma_B = N_B \sigma_B \qquad \ldots \text{(Eq. 7)}$$

where σ_H, σ_A and σ_B are the thermal-neutron absorption cross-sections for hydrogen, and for elements A and B.

The total macroscopic absorption cross-section of the slurry, Σ_T, is given by the sum of Equations 5 to 7, as

$$\Sigma_T = \Sigma_H + \Sigma_A + \Sigma_B \qquad \ldots \text{(Eq. 8)}$$

For a neutron source of a given output, the number of thermal neutrons available for capture by hydrogen in a slurry will be less than those available for capture in pure water by the ratio, $\Sigma_H : \Sigma_T$, because of the neutron-capture reactions with the solid phase. Thus, if P_W and P_S are the prompt-gamma count-rates from hydrogen in pure water and in a slurry, then

$$P_S = \frac{\Sigma_H}{\Sigma_T} P_W \qquad \ldots \text{(Eq. 9)}$$

$$\text{or,} \quad \frac{P_S}{P_W} = f_\sigma = \frac{\Sigma_H}{\Sigma_T} \qquad \ldots \text{(Eq. 10)}$$

By substituting for Σ_H and Σ_T in Equation 10, from the preceding equations, we then obtain

$$f_\sigma = \frac{\left(\frac{W}{M_W} \times 2\sigma_H\right)}{\left(\frac{W}{M_W} \times 2\sigma_H\right) + \left(\frac{S}{M_S} \times Y \times \sigma_A\right) + \left(\frac{S}{M_S} \times Z \times \sigma_B\right)} \qquad \ldots \text{(Eq. 11)}$$

The neutron-capture cross section for oxygen is very small, and the last term in the denominator of Equation 11 may be neglected for oxides.